

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

In re Application of: § BEFORE THE EXAMINER:

Waddell et al.

§ Vickey M. Ronesi

Serial No.: 10/633,001 § Group Art Unit No.: 1714

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For: Elastomeric Composition § Confirmation No.: 8961

Customer No.: 23455 § July 17, 2007

Commissioner for Patents P.O. Box 1450 Alexandria, VA 22313-1450

DECLARATION OF DR. WALTER H. WADDELL UNDER 37 CFR § 1.132

Dear Sir:

- I, Walter H. Waddell, hereby declare that:
- I am a citizen of the United States of America and a resident of the City of Pasadena, TX.
 I am one of the inventors of the subject matter described and claimed in the above-identified patent application, and also in the cited reference WO 02/48257 (Dias).
- 2. I was awarded a Ph.D. degree in chemistry from the University of Houston in May of 1973. I have thirty-five years research and development experience, including as Associate Professor of Chemistry at Carnegie-Mellon University, and Adjunct Professor of Chemical Engineering and Materials Science at The University of Oklahoma, and industry research positions at The Goodyear Tire & Rubber Company, PPG Industries, and ExxonMobil Chemical Co. A copy of my curriculum vitae is attached as Exhibit 1, listing some of my patents and awards, research grants, theses supervised, publications authored, and presentations through 2001.

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- 3. Since May of 1996 I have been continuously employed as a Senior Staff Chemist, Applications Technical Development Manager and Senior Research Associate, by ExxonMobil Chemical Co. conducting research and development work concerning butyl polymers. During this time, I have specifically worked on developing an elastomeric composition comprising isobutylene elastomer, carbon black and polybutene oil described and claimed in the above-identified patent application.
- 4. Under my supervision and control, I provided the data showing the formulations and properties for compositions 1-26 disclosed in Tables 6-14.
- 5. The data were used with off-the-shelf JMP® statistical modeling software commercially available from the SAS Institute to map the effects of various types and amounts of carbon black and types and amounts of polybutene oil, and amounts of naphthenic oil, on the processing characteristics and end use properties of a bromobutyl rubber formulation. The standard formulation reported as composition 1 based on 60 phr N660 carbon black as shown in Dias was used as the model composition for this study.
- 6. A four variable standard rotatable central composite design was selected, and the compound test data for compositions 8-22 appearing in Tables 9-12 of the patent application are shown in the attached table of Exhibit 2 which is captioned N660 Regal 85 017200305. In addition to control runs without any Regal 85/Parapol 1300 (N660/Calsol 810 only) and without any N660/Calsol 810 (Regal 85/Parapol 1300 only), the independent variables and ranges were as follows:

Component	Lower limit, phr	Upper limit, phr
N660 carbon black	20	60
Regal 85 carbon black	40	120
Calsol 810 oil	2	14
Parapol 1300 polybutene	2	14

This four-factor central composite fractional factorial design uses 13 runs or compounds (including 2 duplicate controls) and is nearly as reliable as a full factorial design which may require up to 30 or 40 runs, or more, where each run is laborious and time consuming. The application of such a "design space" approach is standard practice in the art to reduce the quantity of data otherwise required by a full factorial approach.

7. Regression equations were calculated by the JMP® software for the data tabulated in Exhibit 2 using a fit model based on the primary terms only, according to the equation:

Dependent Variable =
$$aW + bX + cY + dZ + constant$$
 (1)

where the terms W, X, Y and Z represent the four independent variables (centered by mean, scaled by range/2) and the coefficients "a" to "d" represent a respective measure of their significance in determining the dependent variable response according to a linear formulation. The JMP output shown in Exhibit 3 indicate that the RSquare values for this model fit are 0.958 for permeability and 0.704 for Mooney Viscosity at 100°C ("MV"), suggesting that the model is qualitative only, especially for MV. The Scaled Estimates (p.1 of Exhibit 3) for permeability are Regal 85 < N660 < 0 < Parapol 1300 < Calsol 810; and for MV are Regal 85 < N660 < 0 < Parapol 1300 < Calsol 810. The trends shown in the Prediction Profiler section of the output (p. 2 of Exhibit 3) indicate that, at the midrange proportions, in general, N660 increases MV and decreases permeability; Regal 85 sharply reduces permeability and sharply increases MV; Calsol 810 sharply increases permeability and has little effect on MV; and Parapol 1300 increases permeability and sharply decreases MV.

8. For a better model, regression equations were calculated by the JMP® software for the data tabulated in Exhibit 2 using a fit model based on both the primary and first order interactive terms according to the equation:

Dependent Variable =
$$aW + bX + cWX + dY + eWY + fXY + gZ + hWZ$$

+ $iXZ + jYZ + Z + constant$ (2)

where the terms W, X, Y and Z represent the four independent variables (centered by mean, scaled by range/2) and the coefficients "a" to "j" represent a respective measure of their significance in determining the dependent variable response according to a linear formulation. This model allows identification of any potential material interactions.

9. The JMP output shown in Exhibit 4 (for the same Exhibit 2 data table) indicate that the RSquare values for the interactive model fit are 0.9789 for permeability and 0.994 for Mooney Viscosity at 100°C ("MV"), indicating that the model of Exhibit 4 is more quantitative than the qualitative model of Exhibit 3, especially for MV. The trends shown

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in the Prediction Profiler section of the output (p. 2 of Exhibit 4) are similar to those of Exhibit 3 except that the effect of N660 on permeability is nearly flat. The coefficients of the interactive terms for the permeability shown in the corresponding Scaled Estimates section are not substantial (the Exhibit 3 model based on the primary terms only already has a comparable RSquare value). On the other hand, for the MV the interactive term coefficients are substantially greater magnitude for Regal 85-Parapol 1300 (-20.7) than for any of N660-Parapol 1300 (-13.9), N660-Calsol 810 (-5.3) or Regal 85-Calsol 810 (-2.96). These results show the pronounced interactive effect of Parapol 1300 and Regal 85 relative to any interaction with N660 and/or Calsol 810.

- 10. The JMP® software is convenient to show predicted changes in the dependent variables by adjusting the independent variables by clicking on and dragging the dashed line representing the phr of each ingredient over the scale of the model in the Predictor Profiler section at the end of page 2 of Exhibits 3 and 4. In Exhibits 3 and 4, the phr is centered at the midrange of the scale of the model. By selecting the phr for each component, the Predictor Profiler will automatically calculate the estimated MV and permeability, read at the left side of the graphs. For example, for the midrange phr of N660 (37.3333 phr), Regal 85 (74.667 phr), Calsol 810 (6.9333 phr) and Parapol 1300 (6.9333 phr), in Exhibit 3 the permeability is predicted at 24.439 and the MV at 54.86. In the more reliable Exhibit 4 model, the midrange-component permeability is again predicted at 24.439, whereas the MV is predicted at 54.86.
- It is also possible to use the JMP® software to set the desired parameters for MV and permeability and have the software calculate the proportions of ingredients to approximate the desired result. For example, in the Prediction Profiler section of the JMP® software output seen in Exhibit 5 (based on the same data table in Exhibit 2), the desirability functions are entered in the right-hand column of graphs. These were entered by mouse and establish low permeability (top curve) and moderate MV (middle curve) as the desired characteristics. The software then calculated the proportions of ingredients to obtain these properties as 0 phr N660, 120 phr Regal 85, 0 phr Calsol 810, and 11.06 phr Parapol 1300, as shown on the bottom of the graphs. The predicted permeability is low at 13.9 and the MV is 61.5 (the setting of the desirability parameter in this example).

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- 12. In the Prediction Profiler of Exhibit 6, the Exhibit 5 model is adjusted to set the proportions of N660 at 60 phr, Regal 85 at 0 phr, Calsol 810 at 0 phr, and Parapol 1300 at 8 phr. In this example, the permeability is estimated at 21, which is better than the 27.47-28.19 for Rows 10 and 11 shown in the table of Exhibit 2 for the 60 phr N660-8 phr Calsol 810 composition. However, the predicted MV in the N660-Parapol 1300 blends of Exhibit 6 is 68.4, which is too high for commercial processing, compared to the 57.6-57.9 for the N660-Calsol 810 blends of Rows 10-11 in Exhibit 2. The Exhibit 6 results indicate that increasing the N660 beyond 60 phr might result in a slightly lower permeability (top-row, left-column graph in the Prediction Profiler), but would result in an even less processable composition with a higher MV (middle-row, left-column graph in the Prediction Profiler).
- 13. Similarly, in Exhibit 7, the Exhibit 6 model is adjusted for 60 phr N660 and 14 phr Parapol 1300, but the MV is still higher than the N660-Calsol 810 controls. Again, the Prediction Profiler (middle-row, left-column graph) indicates that additional N660 would further exacerbate processability by further increasing the 100°C Mooney viscosity. This is consistent with the data for Compositions 1 and 2 in Tables 5 and 6 of the patent application, which explore the effect of N660 black when this is increased from 60 phr to 93.4 with 8 phr Calsol. Increasing the N660 in Composition 2 unacceptably increased the Mooney viscosity and the Shore A Hardness, making the composition too thick for processing and too brittle for use as a tire innerliner. It does not appear possible to increase N660 greater than 60 phr to reduce permeability while at the same time keeping the MV and hardness at suitable levels, even with the addition of oil (Calsol or Parapol).
- 14. In Exhibit 8, the Exhibit 5 model is adjusted to set the proportions of N660 at 0 phr, Regal 85 at 100 phr, and Parapol 1300 at 8 phr. A low permeability (20.2) is predicted, and the MV matches the N660-Calsol 810 controls (54.3). The Prediction Profiler shows (e.g. by setting the N660 at higher amounts using the mouse) that additional N660 can be added with additional Parapol 13 used to offset the higher MV from the N660 with only marginal changes in permeability. Similar results are expected for additional Regal 85. Thus, because the midrange quantity of Parapol 1300 is used in the model with 100 phr Regal 85, an upper limit of 200 phr for the total amount of carbon black (N660 plus Regal

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85) can be estimated for the upper level of Parapol 1300 used in this model – in other words, increases in MV from additional carbon black could still be offset by more Parapol 1300 up to about 200 phr carbon black and 14 phr Parapol 1300.

15. The statistical modeling of Exhibits 2-8 is simplified for the purpose of illustration to 2 dependent variables, permeability and Mooney viscosity. However, other dependent variables must also be considered, such as cure times and physical properties of the cured blend, e.g. scorch values, hardness, etc.

16. In the patent application, compositions 21 and 22 reported in Tables 11 and 12 have processing characteristics and physical properties nearly identical to Compositions 8 and 9, and yet the permeability is much lower. This was not a chance result, because the proportions of ingredients were selected based on the data in Tables 9 and 10 using conventional statistical modeling.

17. I hereby declare that all statements made herein of my own knowledge are true, and that all statements made on information and belief are believed to be true, and further that these statements are made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment or both, under Section 1001 of Title 18 of the United States Code, and that such willful false statements may jeopardize the validity of the application of any patent issuing thereon.

Date July 17, 2007

Walter H. Waddell

Walter HWaddell



EXHIBIT 1

WALTER H. WADDELL

4923 Orange Tree Drive Pasadena, TX 77505 Phone: (281) 998-1811

SUMMARY

Thirty years Research & Development experience using state-of-the-science instrumentation and laboratory procedures to solve applied technical problems and answer scientific questions. Established independent laboratory for fundamental research through external funding as Associate Professor of Chemistry, Carnegie-Mellon University. Successfully directed the research efforts of five doctoral and eight masters degree students, and five postdoctoral research associates. Seven years of experience, including five years of progressive management experience, in three departments as Section Head, Research at The Goodyear Tire & Rubber Company. Directed various programs focused on problem solving in the areas of tire materials and components, textile tire cord adhesion, fabric processing, and tire manufacturing. Directed R&D group as Senior Scientist, Silica R&D at PPG Industries. Developed new applications using precipitated silica in tires and in engineered rubber products, commercialization of new silica products, and improved manufacturing. Co-directed research to surface modify silica to enhance compatibility with polymers as Adjunct Professor of Chemical Engineering and Materials Science at The University of Oklahoma. Used statistical design of experiments and analysis of variance to develop polymers to optimize non-staining tire black sidewall and winter tire tread compound with improved snow and wet traction performance as Applications Technical Development Manager at ExxonMobil Chemical. Currently, Senior Research Associate at ExxonMobil Chemical researching the importance of inflation pressure retention on tire performance, aging and durability.

General accomplishments include selected as a Distinguished Corporate Inventor (1995), recipient of the American Chemical Society, Rubber Divisions' Melvin Mooney Distinguished Technology Award (2003), the Sparks-Thomas Award for outstanding contributions by a young investigator in the field of elastomer chemistry (1993), the Distinguished Service Award (2005), four Best Paper Awards (1994, 1995, 1995, 2000) and five Best Symposium Awards (1994, 1997, 2002, 2004, 2005), recipient of a National Institutes of Health Research Fellowship (1975), being awarded fourteen research grants by foundations and agencies such as the American Chemical Society, National Science Foundation and the National Institute of Health, authoring over one hundred twenty-five publications in scientific journals and in books, presenting over ninety seminars at universities and at scientific meetings, and being an inventor leading to twenty-five patents and five trade secrets.

EDUCATION

RESEARCH ASSOCIATE, Chemistry, Columbia University, New York, NY, 9/73 - 8/75
DOCTOR OF PHILOSOPHY IN CHEMISTRY, University of Houston, Houston, TX, 5/73
BACHELOR OF SCIENCE IN CHEMISTRY, University of Illinois, Chicago, IL, 6/69

EXPERIENCE

ExxonMobil Chemical, Co., Baytown, TX

7/04 – Present
4/00 – 6/04
5/96 - 3/00

SENIOR RESEARCH ASSOCIATE, Butyl Polymers Technology
APPLICATIONS TECHNICAL DEVELOPMENT MANAGER
SENIOR STAFF CHEMIST, Butyl Polymers Technology

Managed technical service group of fourteen developing applications for isobutylene-based elastomers. Optimized non-staining tire black sidewall performance through polymer selection, and developed tread compound formulations with improved winter-traction properties using ExxproTM elastomers. Employed design of experiments and analysis of variance (DOE, ANOVA), surface and near surface characterization techniques (AFM, TEM, Raman) to map polymer blend morphology and filler distribution.

PPG Industries, Inc., Pittsburgh, PA

6/94 - 4/96 SENIOR SCIENTIST, Elastomer Applications, Silica R&D

5/90 - 5/94 SCIENTIST, Elastomer Applications, Silica R&D

Directed research/technical service group of sixteen exploring uses of silicas in rubber, developed surface-modified silicas via joint industrial/university research program, studied fundamentals of silica-polymer interactions and coupling reactions, and merged research and development concepts with customer product goals to establish new applications.

The Goodyear Tire & Rubber Company, Akron, OH

11/89 - 5/90 **SECTION HEAD**, Polymer Chemistry

2/87 - 11/89 SECTION HEAD, Fabric Adhesive & Process Science

2/86 - 1/87 SECTION HEAD, Adhesion & Surface Science

6/83 - 1/86 SENIOR RESEARCH CHEMIST, Tire Compound Chemistry

Managed twenty-two studying structure/property relationships of tire materials, and solving manufacturing problems. Developed predictive tests of cord/compound adhesion, formulated textile adhesives, approved material specifications, streamlined mill processing of tire cords, modified rubber compound surfaces to improve performance, tailored processing equipment surfaces and developed non-fuming additives to improve tire compound physical properties. Defined mechanisms of tire surface ozone aging, reinforcement by in-situ reaction of phenolic resins and silane-silica coupling agents. Explored surface reactions of polymers and metals. Identified non-destructive characterization methods.

Carnegie-Mellon University, Pittsburgh, PA

6/83 - 5/86 ADJUNCT ASSOCIATE PROFESSOR, Chemistry

9/79 - 6/83 **ASSOCIATE PROFESSOR**, Chemistry **ASSISTANT PROFESSOR**, Chemistry

Established research programs studying interactions of UV-visible radiation with reactive organic species (aryl azides, oxygen radicals) and biologically important molecules (visual protein rhodopsin, Vitamin A derivatives) using photochemical, laser spectroscopic, synthetic, cryogenic and chromatographic methods. Trained five postdoctoral associates, and five Ph.D. and eight M.S. graduate students.

PATENTS & AWARDS

- 1. Application of Laser Desorption Mass Spectroscopy to Characterize Rubber Surfaces, Walter H. Waddell, *Goodyear Trade Secret Award*, September, 1984
- 2. Enhanced Adhesion of Rubber to Reinforcing Materials Through the Use of Phenolic Esters, David A. Benko, Syed K. Mowdood, Paul H. Sandstrom, Walter H. Waddell, and Lawson G. Wideman, *United States Patent* 4,605,696, August, 1986
- 3. Reflecting Tire Inner Surface Coating for Post-Cure Tire Analysis, George P. Patitsas, Syed K. Mowdood, Walter H. Waddell, Roop S. Bhakuni and Sharon P. Carmickle, *Goodyear Trade Secret Award*, April, 1988
- 4. Lubricant and Use Thereof for Curing Tires, Syed K. Mowdood, George P. Patitsas and Walter H. Waddell, *United States Patent* 4,780,225, October, 1988
- Improved Epoxy Resin for Polyester/Rubber Composite Adhesion, James G. Gillick, Jimmy L. Richards, Derek Shuttleworth and Walter H. Waddell, Goodyear Trade Secret Award, March, 1989
- 6. Process for the Surface Modification of Unsaturated Rubber by Photochemical Modification with Alkyl Halides, James G. Gillick and Walter H. Waddell, *United States Patent* 4,824,692, April, 1989
- 7. Improved Textile Tire Cord Adhesives Using Reactive Hardeners, James G. Gillick, Jerry L. Brenner, Larry R. Evans, George P. Patitsas, Charles B. Reilly and Walter H. Waddell, *Goodyear Trade Secret Award*, May, 1989
- 8. Rubber Containing Microencapsulated Antidegradants, David A. Benko, Larry R. Evans, James G. Gillick, Walter H. Waddell, Barbara A. Metz, Benjamin F. Benton, Gordon E. Pickett, and Gordon R. Krumm, *United States Patent* 4,895,884, January, 1990
- 9. Concepts for Reduced Tire Weight: Selective Compounding of Tire Innerliner, Daniel F. Klemmensen and Walter H. Waddell, *Goodyear Trade Secret Award*, September, 1990
- 10. Rubber Containing Matrix-Antidegradants, Larry R. Evans, Walter H. Waddell, Frank W. Harris and David A. Benko, *United States Patent* 5,023,287, June, 1991
- Process for the Surface Treatment of Polymers for Reinforcement-to-Rubber Adhesion, Derek Shuttleworth, Syed K. Mowdood, Walter H. Waddell, Jerry L. Brenner and Eilert A. Ofstead, *United States Patent* 5,053,246, October, 1991
- 12. Surface Treating Aluminum Moulds to Improve Release Properties, Cecil Bennett, Derek Shuttleworth and Walter H. Waddell, *European Patent* 374,080, March, 1993
- 13. Metal Oxide Deactivation of Natural Rubber Fatty Acids, David A. Benko, Frederick L. Magnus, Zalman Ronen, Robert W. Strozier and Walter H. Waddell, *United States Patent* 5,254,616, October, 1993

- 14. Process for the Surface Treatment of Polymers for Reinforcement-to-Rubber Adhesion, Jerry L. Brenner, Syed K. Mowdood, Eilert A. Ofstead, Derek Shuttleworth and Walter H. Waddell, *United States Patent* 5,283,119, February, 1994
- 15. Particulate Amorphous Precipitated Silica, James T. Dew, Larry R. Evans and Walter H. Waddell, *United States Patent* 5,353,999, October 1994
- 16. Particulate Amorphous Silica Associated with Thin Polymeric Film, Walter H. Waddell, Larry R. Evans, Jeffrey H. Harwell and John H. O'Haver, *United States Patent* 5,426,136, June, 1995
- 17. Tire Tread Composition Comprising Highly Reinforcing Reinforced Amorphous Precipitated Silica, Larry R. Evans, Walter H. Waddell and Thomas G. Krivak, *United States Patent* 5,605,950, February, 1997
- 18. Tire Tread Composition Comprising Highly Reinforcing Reinforced Amorphous Precipitated Silica, Walter H. Waddell, Larry R. Evans and Thomas G. Krivak, *United States Patent* 5,610,221, March, 1997
- Amorphous Precipitated Silica Having Large Liquid Carrying Capacity, James T.
 Dew, Larry R. Evans, Diana L. Scott and Walter H. Waddell, *United States Patent* 5,906,843, May, 1999
- Transparent and Colorable Elastomeric Compositions, Walter H. Waddell, Robert R. Poulter, Kenneth O. McElrath and John E. Rogers, *United States Patent* 6,624,220, September 23, 2003
- 22. Transparent and Colorable Elastomeric Compositions, Walter H. Waddell, Robert R. Poulter, Kenneth O. McElrath and John E. Rogers, *United States Patent* 6,624,235, September 23, 2003
- 23. Abrasion Resistant Transparent and Colorable Elastomeric Compositions, Walter H. Waddell and Robert R. Poulter, *United States Patent* 6,710,116, March 23, 2004
- 24. Colorable Elastomeric Compositions, Walter H. Waddell and Robert R. Poulter, *United States Patent 6,939,921*, September 6, 2005
- 25. Elastomeric Composition, Walter H. Waddell, Glenn E. Jones, Ilan Duvdevani, United States Patent application 2004/0030036
- 26. Colorable Elastomeric Compositions, Walter H. Waddell and Robert R. Poulter, United States Patent application 2004/0044118
- 27. High Traction and Wear Resistant Elastomeric Compositions, Walter H. Waddell and Robert R. Poulter, *United States Patent application 2004/0063859*
- 28. Elastomeric Composition, Glenn E. Jones, Donald S. Tracey, Walter H. Waddell, *United States Patent application 2004/0087704*

- 29. Elastomeric Blend for Air Barriers Comprising Low Glass Transition Temperature Hydrocarbon Resins, Glenn E. Jones, Mun Fu Tse, Hsien-Chang Wang, Kenneth Lewtas, William M.-T. Chien, Walter H. Waddell, *United States Patent application* 2004/0092648
- 30. Elastomeric Composition, Anthony J. Dias, Glenn E. Jones, Donald S. Tracey, Walter H. Waddell, *United States Patent application 2004/0132894*
- 31. Elastomeric Composition, Walter H. Waddell, David Y.-L. Chung, *United States Patent application 2004/0242731*
- 32. Elastomeric Composition, Walter H. Waddell, David Y.-L. Chung, *United States Patent application* 2004/0242795
- 33. Halogenated Isoolefin Based Terpolymers, Walter H. Waddell, David Y.-L. Chung, United States Patent application 2004/0249085
- 34. Elastomeric Composition, Walter H. Waddell, Donald S. Tracey, and Glenn E. Jones, *United States Patent application 2005/0027062*
- 35. Abrasion Resistant Elastomeric Compositions, Walter H. Waddell and Robert R. Poulter, *United States Patent application 2005/0085594*
- 36. Colorable Elastomeric Compositions, Walter H. Waddell and Robert R. Poulter, United States Patent States Patent application 2005/0137339
- Elastomeric Blend for Air Barriers Comprising Grafted Resin Components, Glenn E. Jones, Mun Fu Tse, Hsien-Chang Wang, Kenneth Lewtas, William M.-T. Chien, Walter H. Waddell, *United States Patent application 2005/0197442*
- 38. Elastomeric Blend for Air Barriers, Glenn E. Jones, Alan G. Galuska, Walter H. Waddell, *United States Patent States Patent application 2005/0222335*
- 39. Innerliners for Use in Tires, Walter H. Waddell, Donald S. Tracey, Stuart W. Botfeld, Dirk F. Rouckhout, *United States Patent application 2006/0167184*

RESEARCH GRANTS

- 1. Spectroscopic Investigation of Retinal Analogs, Research Fellowship Award, National Eye Institute, National Institutes of Health, \$14,500, 9/75-8/76
- 2. Photochemistry of Visual Pigments, *National Society for the Prevention of Blindness*, \$9,000, 2/76-5/78
- 3. Spectral and Photochemical Investigation of Visual Pigments, *Pennsylvania Lions Sight Conservation and Eye Research Foundation*, \$25,100, 5/76-9/82
- 4, Photochemical Investigation of Visual Pigments, *Health Research and Services Foundation*, \$17,358, 7/76-8/78
- 5. ¹³C-NMR Analysis of the Sensitized Photo-oxidations of Polymers, *American Chemical Society, Petroleum Research Fund*, \$10,000, 9/ 7 6-8/7 9
- 6. Protein Chromophore Interactions in Visual Pigments, *National Eye Institute, National Institutes of Health*, \$119,963, 2/77-1/80
- 7. Photochemistry of Rhodopsin and 11-cis-Retinal, *National Eye Institute, National Institutes of Health*, \$28,235, 4/77-3/80
- 8. Superoxide Radicals and Biochemical Hydroxylations, *Health Research and Services Foundation*, \$18,000, 1/78-12/79
- 9. Photoactivated Polymeric Adhesives, *National Science Foundation, Materials Research Laboratory Grant* to Carnegie-Mellon University, \$93,513, 7/78-6/81
- Purchase of a UV-Visible Spectrometer, National Science Foundation, \$20,000, 8/79-1/81
- Superoxide Radicals and Biochemical Hydroxylations, National Institutes of Neurological and Communicative Disorder and Stroke, National Institutes of Health, \$149,827, 12/79-11/82
- 12. Dopamine-beta-Hydroxylase and the Superoxide Radical, *Western Pennsylvania Heart Association*, \$7,500, 7/80-6/81
- 13. Photochemistry of Linear Polyenes Related to the Visual Chromophore, *American Chemistry Society, Petroleum Research Fund*, \$30,500, 9/80-8/82
- 14. Spectroscopic Analysis of Laser Activated Bond Formation in Polymeric Adhesives, National Science Foundation, Materials Research Laboratory Grant to Carnegie-Mellon University, \$71,580, 7/81-6/83

THESES SUPERVISED

- 1. The trans-->cis Photoisomerization of all-trans-Retinal and Some Synthetic Analogs, Daniel Lee Hopkins, *Master of Science*, 1978
- 2. Synthesis and Photochemistry of Some Platinum (II)-Alkoxycarbonyl Complexes, Conrad Frank Shiba, *Doctor of Philosophy*, 1979
- 3. Influence of Alkyl Substitution on the trans-->cis Photoisomerization of all-trans-Retinal and Related Polyenes, John Lawton West, *Master of Science*, 1979
- 4. The Influence of Methyl Substitution on the Photochemistry of Retinal, John Lawton West, *Doctor of Philosophy*, 1980
- 5. Qualitative Studies of the Low Temperature Photochemistry of Rhodopsin and Related Pigments, Juliette Lecomte, *Master of Science*, 1980
- 6. The Photoinitiated Decomposition of Phenyl Azide and Its Derivatives. Evidence for a Chain Reaction, Celia H Lee Go, *Doctor of Philosophy*, 1982
- A Time-Resolved Assay of Dopamine-beta-Hydroxylase Activity Utilizing High-Pressure Liquid Chromatography, Natalie Barbara Feilchenfeld, Master of Science, 1982
- 8. Reactive Species Produced by the 5-Methylphenazinium Methylsulfate/Reduced beta-Nicotinamide Adenine Dinucleotide/02 System in the Hydroxylation of Benzoic Acid, Rachel Ellen Lewis, *Master of Science*, 1982
- 9. Synthesis and Solution Photochemistry of all-trans-1,1,2-Trideuterio-octatetraene, Roseann Frances McDaniel, *Master of Science*, 1983
- Pulsed-Laser and Cryogenic Spectroscopic Investigations of Intermediates Formed in the Photo-initiated Autocatalytic Chain Decomposition (PACD) Reaction of Phenyl Azide and Phenyl Isocyanate, Natalie Barbara Feilchenfeld, *Doctor of Philosophy*, 1983
- 11. The trans-->cis Photoisomerization of Protonated Retinyl Schiff Bases, Jean Marie Donahue, *Master of Science*, 1984
- 12. Kinetic Analysis of the Photoinitiated Autocatalytic Chain Decomposition of Phenyl Azide. A Molecular Explosion in Solution, Joseph Pascaul Costantino, *Master of Science*, 1984
- Synthesis and Photochemistry of Reactive Organic Compounds: Part I Octatetraene and Its Deuterated Analogue. Part II Phenyl Azide and Its
 Substituted Derivatives, Roseann Frances McDaniel, Doctor of Philosophy, 1986

PUBLICATIONS

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- 2. Spectroscopy and Photochemistry of Rhodopsin Models: Retinals, Retinal Schiff Bases and Protonated Retinal Schiff Bases, Walter H. Waddell, *Dissertation Abstracts Int B*, **34 (4)**, 1437 (1973)
- 3. Visual Pigments III. Determination and Interpretation of the Fluorescence Quantum Yields of Retinals, Schiff Bases and Protonated Schiff Bases, Walter H. Waddell, Arnold M. Schaffer and Ralph S. Becker, *Journal of the American Chemical Society*, **95**, 8223-8227 (1973)
- 4. Visual Pigments IV. Experimental and Theoretical Investigations of the Absorption Spectra of Retinal Schiff Bases and Retinals, Arnold M. Schaffer, Walter H. Waddell and Ralph S. Becker, *Journal of the American Chemical Society*, **96**, 2063-2068 (1974)
- 5. Relative Emission Efficiencies of Deuterated Benzenes, Walter H. Waddell, Carl A. Renner and Nicholas J. Turro, *Molecular Photochemistry*, **6**, 321-324 (1974)
- 6. Quantum Chain Processes. Direct Observation of High Quantum Yields in the Direct and Photosensitized Excitation of Tetramethyl-1,2-Dioxetane, Nicholas J. Turro and Walter H. Waddell, *Tetrahedron Letters*, **25**, 2069-2072 (1975)
- 7. Energy Storage and Release. Direct and Sensitized Photoreactions of Benzvalene. Evidence for a Quantum Chain Process, an Adiabatic Photorearrangement, a Degenerate Photovalence Isomerization and Two Reactive Triplet States, Carl A. Renner, Thomas J. Katz, Joseph Pouliquen, Nicholas J Turro and Walter H. Waddell, *Journal of the American Chemical Society*, 97, 2568-2570 (1975)
- 8. Photoluminescence of Bicyclo(2.2.1)Heptan-2-one (Norcamphor) and Related Molecules, Walter H. Waddell, Nicholas J. Turro and George Farrington, *Molecular Photochemistry*, **7**, 475-497 (1976)
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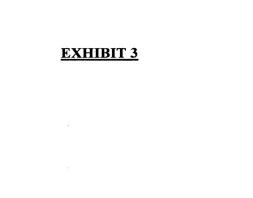
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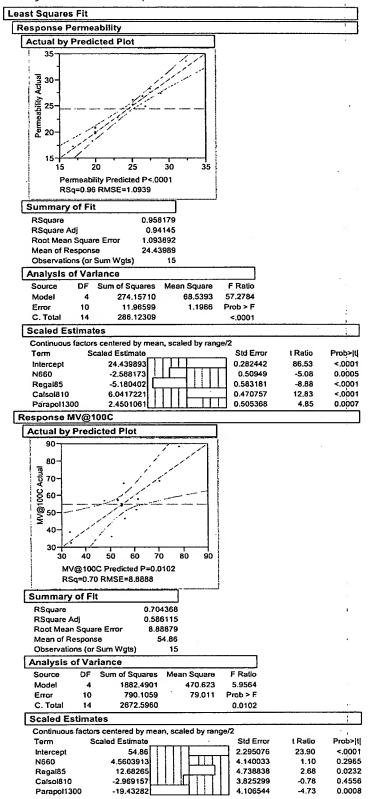
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EXHIBIT 2

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40 80 14 32.3 17.23 3.79 5.92 4.72 8.64 36.5 8.12 1.96 924 60 80 14 2 68.2 11.82 8.7 3.29 4.45 8.74 62.7 7.41 7.05 9.83 5.07 4.63 8.74 7.41 7.05 9.83 5.07 4.63 8.74 7.41 7.05 3.51 9.83 5.07 6.64 4.07 8.01 2.48 8.45 9.81 6.74 4.07 8.01 2.48 8.45 9.74 7.41 7.05 3.74 6.41 9.81 6.07 6.64 9.75 9.81 6.07 6.64 9.75 9.81 6.07 6.64 9.75 9.81 6.75 9.75		40		2	8	6.99		7.96		4.53	8.87	59.7	7.82	5.45	658	17.87	20.0626
60 80 14 87 4.5 8.74 62.7 7 5.98 513 60 120 12 14 35.9 10.37 9.88 3.04 4.33 8.19 72.9 7.41 7.05 35.1 20 120 2 14 38.5 10.37 9.88 3.04 4.33 8.19 72.9 7.41 7.05 35.1 20 120 12 4.0 8.56 40.7 8.01 6.24 9.24 6.74 6.75 6.54 9.05 7.0 40 4.0 8 4.6 4.12 4.76 8.26 50.7 6.54 4.05 8.74 6.74 4.03 8.45 8.05 6.74 6.74 4.03 8.45 8.05 8.07 6.54 4.03 8.05 8.05 8.07 6.54 4.03 8.05 8.05 8.05 8.05 8.05 8.05 8.05 8.05 8.05 8.05 8.05 <td>1 (*</td> <td>40</td> <td></td> <td>14</td> <td>14</td> <td>32.3</td> <td></td> <td>3.79</td> <td></td> <td>4.72</td> <td>8.64</td> <td>36.5</td> <td>8.12</td> <td>1.96</td> <td>924</td> <td>13.74</td> <td>33.4616</td>	1 (*	40		14	14	32.3		3.79		4.72	8.64	36.5	8.12	1.96	924	13.74	33.4616
60 120 2 14 35.9 10.37 9.88 3.04 4.33 8.19 72.9 7.41 7.05 351 20 80 80 80 40.7 8.01 2.48 845 20 120 40 80 50.7 4.63 8.56 40.7 8.01 2.48 845 60 40 40 4 4.12 4.76 8.96 50.7 6.54 7.96 8.91 6.74 6.41 6.41 6.41 4.02 7.98 7.98 8.96 6.74 6.74 6.71 6.74 6.41 4.03 8.96 50.7 6.54 4.03 8.96 6.74 6.74 4.03 8.96 6.74 6.74 4.03 8.96 6.74 6.74 4.03 8.96 6.74 4.03 8.96 6.74 4.03 8.96 6.74 4.03 8.96 6.74 4.04 8.96 4.75 4.76 8.76 4.77 <t< td=""><td>4</td><td>09</td><td></td><td>14</td><td>2</td><td>68.2</td><td></td><td></td><td></td><td>4.45</td><td>8.74</td><td>62.7</td><td>7</td><td>5.98</td><td>513</td><td>9.67</td><td>26.8082</td></t<>	4	09		14	2	68.2				4.45	8.74	62.7	7	5.98	513	9.67	26.8082
20 80 8 14 38.5 16 4.55 5.07 4.63 8.56 40.7 8.01 2.48 845 20 120 14 8 4.55 5.07 4.63 8.96 50.7 6.54 3.74 641 60 40 4 4.0 4.12 4.76 8.96 50.7 6.54 7.96 7.98 7.91	2	9		2	14	35.9	·			4.33	8.19	72.9	7.41	7.05	351	5.43	17.2323
20 120 14 8 46.4 14.28 6.44 4.12 4.76 8.96 50.7 6.54 3.74 641 60 40 8 56.8 14.55 6.59 3.7 4.32 8.2 52.3 7.98 4.26 7.09 40 120 8 16.03 10.99 2.8 4.31 8.81 6.91 6.74 6.44 4.03 60 0 57.9 13.13 6.24 3.59 4.67 10.84 3.68 836 60 0 57.9 13.13 6.24 3.59 4.67 10.84 3.68 836 60 100 57.6 12.67 6.25 3.52 3.98 8.66 45.9 11.04 3.71 865 70 100 57.6 13.18 6.28 3.99 4.47 8.91 4.85 9.11 3.39 804 80 80 80 8.53 13.64	9	20				38.5			-	4.63	8.56	40.7	8.01	2.48	845	16.45	27.3238
60 40 8 56.8 14.55 6.59 3.7 4.32 8.2 52.3 7.98 4.26 709 40 120 8 16.03 10.99 2.8 4.31 8.81 69.1 6.74 6.44 403 60 0 8 0 57.9 13.13 6.24 3.58 4.67 46.7 10.84 6.44 403 60 0 57.9 13.13 6.24 3.58 4.67 46.7 10.84 3.68 836 10 10 8 53.7 13.12 6.24 3.59 8.66 45.9 11.04 3.71 865 10 10 8 53.7 13.12 6.14 40.7 44.7 9.13 3.59 775 10 10 8 54.9 13.86 3.99 4.54 8.91 45.5 9.11 3.59 4.54 8.91 4.55 9.11 9.13 9.13 <th< td=""><td>1</td><td>200</td><td>Ì</td><td>14</td><td>8</td><td>46.4</td><td></td><td>1</td><td></td><td>4.76</td><td>8.96</td><td>50.7</td><td>6.54</td><td>3.74</td><td>641</td><td>13.08</td><td>28.4259</td></th<>	1	200	Ì	14	8	46.4		1		4.76	8.96	50.7	6.54	3.74	641	13.08	28.4259
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0 100 0 8 54.9 13.08 6.28 3.99 4.42 8.91 48.5 9.11 3.39 804 2 40 80 8 8 56.9 13.37 6.7 3.86 4.54 8.91 53.5 7.45 4.49 669 1 40 80 8 8 57.4 13.82 7.19 3.81 4.65 8.98 53.9 7.43 4.54 650	2	0		0	8	53.7				4.42	8.93	47.7	9.13	3.59	775	20.55	19.7465
40 80 8 8 56.9 13.37 6.7 3.86 4.54 8.91 53.5 7.45 4.49 669 1 40 80 8 6 7.19 3.81 4.65 8.98 53.9 7.43 4.54 650		0	1	0	8	54.9			3.99	4.42	8.91	48.5	9.11	3.39	804	20.32	20.7516
40 R0 R R 8 57.4 13.82 7.19 3.81 4.65 8.98 53.9 7.43 4.54 650	4	40	-						3.86	4.54	8.91	53.5	7.45	4.49	699	13.77	25.2106
	7	40		8	8	57.4	13.82	7.19	3.81	4.65	8.98	53.9	7.43	4.54	. 650	13.8	26.0908





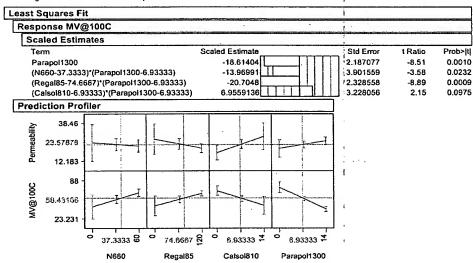
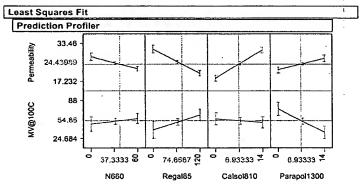
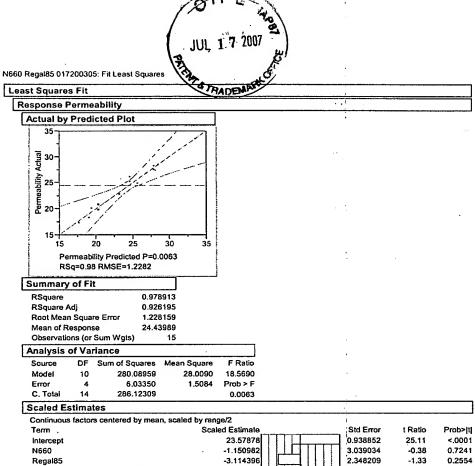


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(N660-37.3333)*(Regal85-74.6667) 0.0591635 1.532426 0.04 0.9711 5.566476 2.505293 2.22 0.0904 Calsol810 (N660-37.3333)*(Calsol810-6.93333) 1.8343251 2.173488 0.84 0.4462 (Regal85-74.6667)*(Calsol810-6.93333) 1.3892384 5.020086 0.28 0.7957 1.377765 0.1338 Parapol1300 2.5858517 1.88 (N660-37.3333)*(Parapol1300-6.93333) (Regal85-74.6667)*(Parapol1300-6.93333) 0.8283 2.457816 -0.23 -0.568979 0.1869 2.3330606 1.466892 1.59 (Calsol810-6.93333)*(Parapol1300-6.93333) 1.5542149 0.4873

ĸe	sponse	3 M	<u>ข@าเ</u>	JUC				
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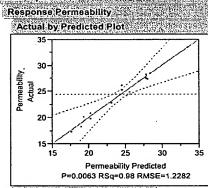
Summary of Fit	
RSquare	0.994311
RSquare Adj	0.98009
Root Mean Square Error	1.94959
Mean of Response .	54.86
Observations (or Sum Wgts)	15

Analysis	of Va	riance		
Source	DF	Sum of Squares	Mean Square	F Ratio
Model	10	2657.3924	265.739	69.9148
Error	4	15.2036	3.801	Prob > F
C. Total	14	2672.5960		0.0005

Scaled Estimates			1		
Continuous factors centered by mean, scale	d by range/2		;	•	
Term	Scaled Estimate		Std Error	t Ratio	Prob> t
Intercept	58.431657		1.490342	39.21	<.0001
N660	11.923302		4.82419	2.47	0.0688
Regal85	10.630306		3.727568	2.85	0.0463
(N660-37.3333)*(Regal85-74.6667)	-3.361981		2.432587	-1.38	0.2391
Calsol810	-12.4358		13.976925	-3.13	0.0353
(N660-37.3333)*(Calsol810-6.93333)	-5.302749		3.450214	-1.54	0.1991
(Regal85-74.6667)*(Calsol810-6.93333)	-2.963747	<u> </u>	7.968931	-0.37	0.7288

EXHIBIT 5

Least Squares Fit



Summary of Fit

RSquare	0.978913
RSquare Adj	0.926195
Root Mean Square Error	1.228159
Mean of Response	24.43989
Observations (or Sum Wgts)	15

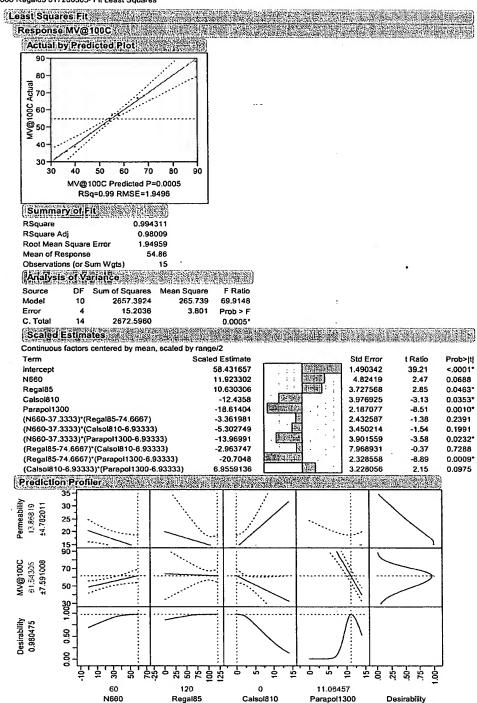
Analysis of Variance

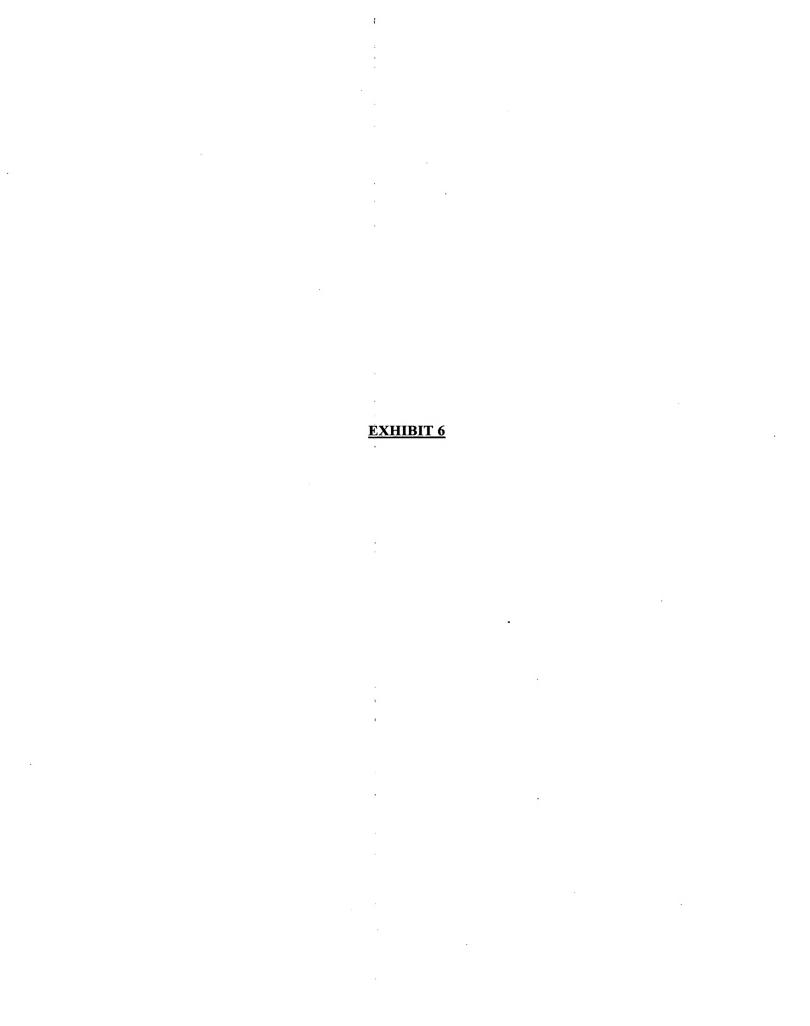
Source	DF	Sum of Squares		F Ratio
Model	10	280.08959	28.0090	18.5690
Error	4	6.03350	1.5084	Prob > F
C. Total	14	286.12309		0.0063*

Continuous	factors	centered	by mean.	scaled	by range/2

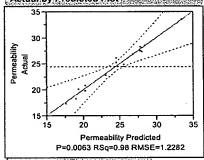
Commission received by mean, comme	-, .ag., -
Tem	Scaled Estimate
Intercept	23.57878
N660	-1.150982
Regal85	-3.114396
Calsol810	5.566476
Parapol1300	2.5858517
(N660-37.3333)*(Regal85-74.6667)	0.0591635
(N660-37.3333)*(Calsol810-6.93333)	1.8343251
(N660-37.3333)*(Parapol1300-6.93333)	-0.568979
(Regal85-74.6667)*(Calsol810-6.93333)	1.3892384
(Regal85-74.6667)*(Parapol1300-6.93333)	2.3330606
(Calsol810-6.93333)*(Parapol1300-6.93333)	1.5542149

	; Std Error	t Ratio	Prob> t
- 1 4 4 4 4 4 4 4 4 4 4 4 4 4 4 4 4 4 4	0.938852	25.11	<.0001*
E	3.039034	-0.38	0.7241
- 25 TH	2.348209	-1.33	0.2554
1	2.505293	2.22	0.0904
3021	1.377765	1.88	0.1338
	1.532426	0.04	0.9711
(数)	2.173488	0.84	0.4462
	2.457816	-0.23	0.8283
銀	· 5.020088	0.28	0.7957
	1.466892	1.59	0.1869
1 1	2 022527	0.76	0.4972





Actual by Predicted Plot



Summary of Fit	
RSquare	0.978913
RSquare Adj	0.926195
Root Mean Square Error	1.228159
Mean of Response	24.43989
Observations (or Sum Wgts)	15

Source	Ur	Sum or Squares	, incan oquale	1 11000
Model	10	280.08959	28.0090	18.5690
Error	4	6.03350	1.5084	Prob > F
C. Total	14	286.12309		0.0063*

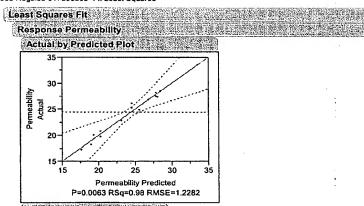
Scaled Estimates Continuous factors centered by mean, scaled by range/2

Term Scaled Estimate Intercept 23.57878 -1.150982 -3.114396 5.566476 N660 Regal85 Calsol810 Parapol1300 2.5858517 (N660-37.3333)*(Regal85-74.6667) (N660-37.3333)*(Calsol810-6.93333) 0.0591635 1.8343251 (N660-37.3333)*(Parapol1300-6.93333) -0.568979 (Regal85-74.6667)*(Calsol810-6.93333) (Regal85-74.6667)*(Parapol1300-6.93333) (Calsol810-6.93333)*(Parapol1300-6.93333) 1.3892384 2.3330606 1.5542149

			4.		
			Std Error	t Ratio	Prob> t
	1 1	建加料 机	0.938852	25.11	<.0001*
	× 1	1	3.039034	-0.38	0.7241
	200		2.348209	-1.33	0.2554
		20000000000000000000000000000000000000	2.505293	2.22	0.0904
		28.0	1.377765	1.88	0.1338
l		- 2	1.532426	0.04	0.9711
		1 1 1 1 1 1 1 1 1 1 1 1 1 1 1 1 1 1 1	2.173488	0.84	0.4462
	[]	_ : !	2.457816	-0.23	0.8283
l	- : "	46	5.020086	0.28	0.7957
l		78	1.466892	1.59	0.1869
Ŀ		級	2.033537	0.76	0.4873

Least Squares Fit Response MV@100C Actual by Predicted Plot 80 Actual 70 260 ® 50 30 40 50 60 70 80 30 MV@100C Predicted P=0.0005 RSq=0.99 RMSE=1.9496 Summary of Fit 0.994311 RSquare 0.98009 RSquare Adj Root Mean Square Error 1.94959 Mean of Response 54.86 Observations (or Sum Wgts) 15 Analysis of Variance Sum of Squares F Ratio Source DF Mean Square 2657.3924 265.739 69.9148 Model 10 15.2036 3.801 Error Prob > F C. Total 2672.5960 0.0005* Scaled Estimates Continuous factors centered by mean, scaled by range/2 Std Error Scaled Estimate t Ratio Prob>|t| Term 58.431657 1.490342 39.21 <.0001 Intercept N660 11.923302 400 4.82419 2.47 0.0688 Regal85 10.630306 3.727568 2.85 0.0463* Calsol810 -12.4358 3.976925 -3.13 0.0353* Parapol1300 -18.61404 2.187077 -8.51 0.0010* (N660-37.3333)*(Regal85-74.6667) (N660-37.3333)*(Calsol810-6.93333) (N660-37.3333)*(Parapol1300-6.93333) 2.432587 -1.38 0.2391 -3.361981 -5.302749 3.450214 -1.54 0.1991 -13.96991 3.901559 -3.58 0.0232* (Regal85-74.6667)*(Calsol810-6.93333) -2.963747 7.968931 -0.37 0.7288 (Regal85-74.6667)*(Parapol1300-6.93333) -20.7048 2.328558 -8.89 0.0009 (Calsol810-6.93333)*(Parapol1300-6.93333) 6.9559136 3.228056 2.15 0.0975 Prediction Profiler 35 Permeability 21.00691 ±19.47925 30-25 , 20 15 90 MV@100C 68,43585 ±30,92154 70 50-30. Desirability 0.621151 0.50 8 225 -62. 60 N660 0 0 Regal85 Calsol810 Parapol1300 Desirability

EXHIBIT 7



RSquare	0.978913
RSquare Adj	0.926195
Root Mean Square Error	1.228159
Mean of Response	24.43989
Observations (or Sum Wgts)	15

Analysi	SOLV	atiance		AT THE PARTY.
Source	DF	Sum of Squares	Mean Square	F Ratio
Model	10	280.08959	28.0090	18.5690
Error	4	6.03350	1.5084	Prob > F
C. Total	14	286.12309		0.0063*

Scaled Estimates Continuous factors centered by mean, scaled by range/2

Term	Scaled Estimate
Intercept	23.57878
N660	-1.150982
Regal85	-3.114396
Calsol810	5.566476
Parapol1300	2.5858517
(N660-37.3333)*(Regal85-74.6667)	0.0591635
(N660-37.3333)*(Calsol810-6.93333)	1.8343251
(N660-37.3333)*(Parapol1300-6.93333)	-0.568979
(Regal85-74.6667)*(Calsol810-6.93333)	1.3892384
(Regal85-74.6667)*(Parapol1300-6.93333)	2.3330606
(Calsol810-6.93333)*(Parapol1300-6.93333)	1.5542149

	Std Error	t Ratio	Prob> t
. 人名英拉拉	0.938852	25.11	<.0001*
	3.039034	-0.38	0.7241
	2.348209	-1.33	0.2554
	2.505293	2.22	0.0904
202	1.377765	1.88	0.1338
1	1.532426	0.04	0.9711
	2.173488.	0.84	0.4462
	2.457816	-0.23	0.8283
	5.020086	0.28	0.7957
	1.466892	1.59	0.1869
: i 🎉	2.033537	0.76	0.4873

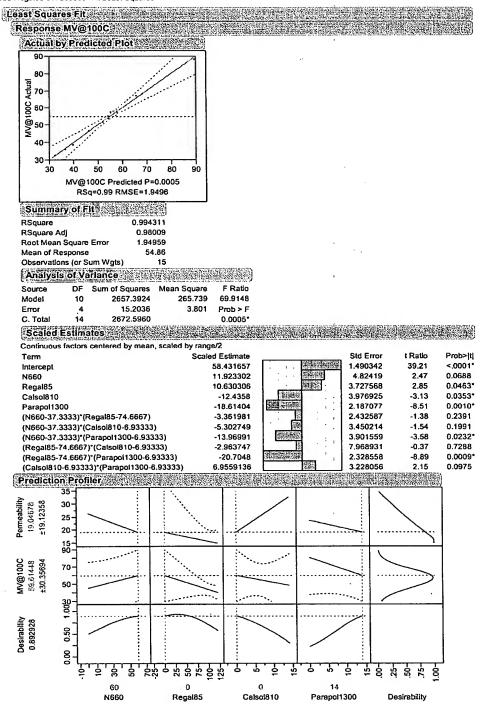
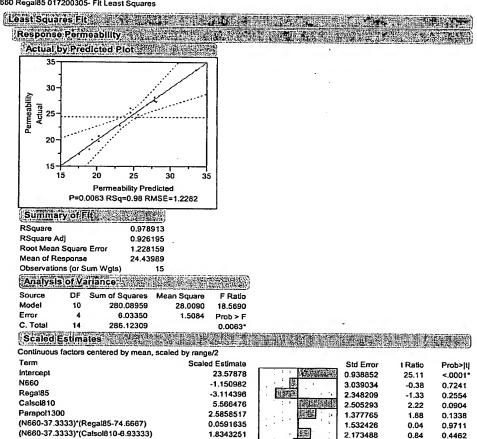


EXHIBIT 8

(N660-37.3333)*(Parapol1300-6.93333) (Regal85-74.6667)*(Calsol810-6.93333) (Regal85-74.6667)*(Parapol1300-6.93333)

(Calsol810-6.93333)*(Parapol1300-6.93333)



-0.568979

1.3892384

2 3330606

1.5542149

2.457816

5.020086

1.466892

2.033537

-0.23

0.28

1.59

0.76

0.8283

0.7957

0.1869

0.4873

N660

Regal85

Calsol810

Parapol1300

Desirability

Least Squares Fit Response MV@100C Actual by Predicted Plot 80 Actual 70 200 © 50 60 70 80 40 50 30 MV@100C Predicted P=0.0005 RSq=0.99 RMSE=1.9496 Summary of Fit 0.994311 RSquare RSquare Adj 0.98009 Root Mean Square Error 1.94959 Mean of Response 54.86 Observations (or Sum Wgts) 15 Analysis of Variance Sum of Squares F Ratio DF Mean Square Source 2657.3924 265.739 69.9148 Model 10 15.2036 3.801 Prob > F Error C. Total 2672.5960 0.0005* Scaled Estimates Continuous factors centered by mean, scaled by range/2 Std Error t Ratio Prob>Iti Term Scaled Estimate 58.431657 1,490342 39.21 <.0001 Intercept N660 11.923302 4.82419 0.0688 Regal85 10.630306 3.727568 2.85 0.0463* Calsol810 -12.4358 3.976925 -3.13 0.0353* 2.187077 0.0010 Parapol1300 -18.61404 -8.51 (N660-37.3333)*(Regal85-74.6667) (N660-37.3333)*(Calsol810-6.93333) (N660-37.3333)*(Parapol1300-6.93333) 0.2391 -3.361981 2.432587 -1.38-5.302749 3.450214 -1.54 0.1991 -13.96991 3.901559 -3.58 0.0232* (Regal85-74.6667)*(Calsol810-6.93333) -2.963747 7.968931 -0.37 0.7288 (Regal85-74.6667)*(Parapol1300-6.93333) -20.7048 2.328558 -8.89 0.0009* (Calsol810-6.93333)*(Parapol1300-6.93333) 6.9559136 3.228056 2.15 0.0975 Prediction Profiler 35 Permeability 20.24905 ±2.411174 30 25 20-15 90 MV@100C 54.3 ±3.82752 70 50-Desirability 0.857663 0.50 0.00 2 2 5 5 5 용 5 ₽ -25. -02. -27. -09. 100 G 0 8